

### Abstract

The present invention is a process for the preparation of 17 $\beta$ -hydroxy-7 $\alpha$ -methyl-19-nor-17 $\alpha$ -pregn-5(10)-en-20-yn-3-one (17 $\alpha$ -ethynyl-17 $\beta$ -hydroxy-7 $\alpha$ -methyl-5(10)-estren-3-one, tibolone) of formula 1, which comprises hydrolysis of 17 $\alpha$ -ethynyl-17 $\beta$ -hydroxy-7 $\alpha$ -methyl-5(10)-estrene 3,3-cyclic ketals of formula 2, where groups R<sub>1</sub>, R<sub>2</sub>, R<sub>3</sub> and R<sub>4</sub> are hydrogen atoms or alkyl groups, or R<sub>1</sub> and R<sub>3</sub>, taken together with the carbon atoms within the dioxolane ring to which they are attached, form an alicyclic ring fused to the dioxolane ring, with R<sub>2</sub> and R<sub>4</sub> being hydrogen atoms, or R<sub>1</sub> and R<sub>3</sub> together with the carbon atoms to which they are attached form an aromatic ring fused to the dioxolane ring, where R<sub>2</sub> and R<sub>4</sub>, taken together, form a chemical bond within said aromatic ring. In addition, the present invention includes an intermediate, compound of formula 2 and two processes to prepare 17 $\alpha$ -ethynyl-17 $\beta$ -hydroxy-7 $\alpha$ -methyl-5(10)-estrene 3,3-cyclic ketals of formula 2: (a) by contacting 17 $\alpha$ -ethynyl-17 $\beta$ -hydroxy-7 $\alpha$ -methyl-4-estren-3-one with vicinal diols in the presence of a protic acid, and (b) by contacting 7 $\alpha$ -methyl-5(10)-estrene-17-one 3,3-cyclic ketals of formula 4, where R<sub>1</sub>-R<sub>4</sub> are defined as above, with metal acetylides, in inert solvents.